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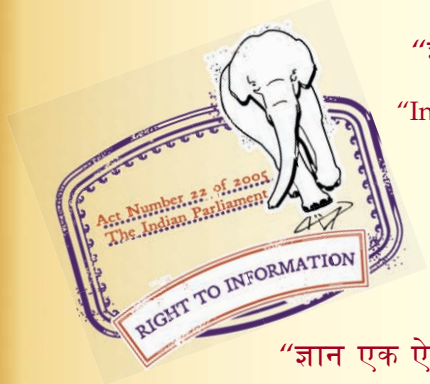
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IS 13624 (1993): Methods of Sampling and Analysis of Water Formed Deposits--Guidelines. UDC 663.6 : 543.3

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जल निर्मित निक्षेपों के नमूने लेने और विश्लेषण की पद्धतियों के सिद्धान्त – मार्गदर्शिका

Indian Standard

METHODS OF SAMPLING AND ANALYSIS OF WATER FORMED DEPOSITS — GUIDELINES

UDC 663·6 : 543·3

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NEW DELHI 110002

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Price Group 7

Water Sectional Committee, CHD 013

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Sectional Committee had been approved by the Chemical Division Council.

In industry, water is used for various purposes and comes in contact with different materials of constructions either in liquid or gaseous state at different temperatures. When the surfaces of the materials of construction or equipment get fouled with scales, corrosion products, biological deposits or sludges, may lead to their premature failure. These products are either formed by the undesirable soluble or insoluble contents carried over by the water from the source or formed by the reaction of water or its contents with surfaces in contact with it.

Examination and analysis of these water formed deposits will provide sufficient information to diagnose the reasons of formation of such deposits and would help to take corrective steps such as suitable treatment of water before its use and prevention of pollution of either water or the environment, etc. The analysis will further help for evolution of methods for removing these deposits easily without jeopardising the further utility of the materials and equipment.

In the preparation of this standard considerable assistance has derived from ASTM.

In reporting the test result or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it should be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF SAMPLING AND ANALYSIS OF WATER FORMED DEPOSITS — GUIDELINES

1 SCOPE

1.1 This standard covers the types of deposits on equipment surfaces in steam-water system and the methods of sampling for analysis and identification of their constituent describing briefly the nature of the deposits.

1.1.1 Attempt has been made to frame the guidelines for presentation of those methods that shall be suitable for most deposits, if not all. Methods of sampling may, however, be conveniently replaced or modified with improved techniques as they become available from time to time. However, it is important that sampling devices capable of giving a representative sample shall be used.

1.1.2 These guidelines are based on the experience gained over the years, with only those water formed deposits which are encountered while using water in the industry involving heat exchangers, steam raising equipment (boiler and other accessories) and steam rotating equipment with accessories, which include the end use of the steam.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard :

IS No.	*Title*
460 (Part 1) : 1978	Specification for test sieve : Part 1 Wire cloth test sieve (*second revision*)
1917 : 1962	Methods of chemical analysis of quartzite and high silica sand
5194 : 1969	Methods of determination of nitrogen — Kjeldale method
6361 : 1971	Methods of colorimetric determination of phosphates

3 TYPES OF DEPOSITS

3.1 Each water formed deposit may have different characteristics depending upon the particular processing, conditions of use of water in the process, materials of constructions in contact with water, temperature and pressure of the system and overall quality of water used in the process. For example, in heat exchangers, general form of deposits include:

a) sediments of the suspended materials present in the water under use,

b) erosion products of the materials,

c) hardness scales consisting of calcium carbonate, magnesium hydroxides, calcium sulphate due to (temporary and permanent) hardness present in water, and

d) metal oxides and hydroxides due to corrosion of the materials, etc. On the other hand, the deposits on the turbine blades may generally be of silica, or complex silicates, a very hard and adherent scale and is quite difficult to remove. If the water quality or the steam quality together with details of process are known, there may not be any difficulty in predicting the nature of deposits formed in any section of the system.

However, in most of the industries including the power stations (nuclear and thermal) the steam-water cycle is quite large and there is a full fledged possibility of mass transport involving transfer of deposits from one part to the other part of the system. Hence detailed analysis of the individual deposits are the only way to know the causes of deposits and to take corrective action accordingly.

3.2 Table 1 illustrates the location of certain substances or mineral species in boilers.

Table 1 Location of Mineral Species in Boilers

(*Clause* 3.2)

Sl No.	Nature of Compound	Mineral Species	Areas Where Found
1.	Carbonates of alkaline earth and heavy metals	Aragonite ($CaCO_3$) Calcite ($CaCO_3$) Dolomite ($CaCO_3$ Mg CO_3) Ankerite ($CaCO_3$) (Fe, Mg, $MnCO_3$) Gaylusite ($CaCO_3$) Na_2Co_3. $5H_2O$	In heat exchange, softener and boil sludges due to precipitation of bicarbonates under heat or high *p*H influence. In low pressure boilers. Precipitate under high heat influence
2.	Hydroxides of alkaline earth and heavy metals	Brucite [Mg (OH)$_2$]	In boiler and softener sludges, due to hydrolysis under high *p*H conditions
		Lime [Ca (OH)$_2$]	In lime slurry dosing line and in steam line due to thermal hydrolysis previously deposit calcium carbonate.
		Ferrous [Fe (OH)$_2$] hydroxide Ferric [Fe (OH)$_3$] hydroxide	In corrosion pits to reaction of metal with water
		Nickel hydroxide [Ni (OH)$_2$]	In cupronickel stage heaters due to exfoliation.
3.	Metal oxides and compounds of metals under contact with water	Haematite (Fe_2O_3) Magnetite (Fe_3O_4) Iron oxide (FeO) Cuprite (Cu_2O) Tenorite (CuO)	In boiler sludges on tube surfaces to corrosion by due water/steam
		Corundum 6 (— Al_2O_3) Zincite (ZnO)	In boiler sludges and on turbines due to material corrosion
		Atacamite 6 [— $CuCl_2$. $3Cu$ (OH)$_2$] Azurite [$2CuCO_3$. Cu (OH)$_2$] Brochantite [$CuSO_4$. $3Cu$ (OH)$_2$]	On metallic surfaces and on corrosion products
4.	Phosphates of alkali and alkaline earth metals and iron	Hydroxyapatite [Ca_{10} (OH)$_2$ (PO_4)$_6$]	In boiler sludge due to treatment chemicals
		Wilkeite [$Ca_{10}O$ (Si, P, S)O_4]$_6$ Sodium calcium phosphate $NaCa$ (PO_4) Whitlockite (B-Ca_3 P_2 O_8) Dicalcium phosphate ($CaHPO_4$)	In boiler scale due to high concentration of treatment chemicals and in boiler sludges
		Magnesium hydroxyphosphate [Mg_3 PO_4. Mg (OH)$_2$]	In boiler sludge due to high phosphate low silica concentration.
		Vivianite [Fe_3 (PO_4)$_2$. $8H_2O$]	On cooling system surfaces due to phosphate dose
5.	Silica and silicates	Cristobalite (SiO_2)	In boiler scale and turbine scale due precipitation of silicic acid and carry-over in steam respectively
		Free Silica (SiO_2)	On heat exchanger surfaces due to deposition of sand or dirt in cooling water
		Surpentine ($3MgO$. $2SiO_2$. $2H_2O$) Talc (3 MgO. 4 SiO_2. H_2O) Sepiolite ($2MgO$. $3SiO_2$. $2H_2O$)	In boiler sludge due to reaction of Mg (OH)$_2$ with silica

Table 1 (*concluded*)

Sl No.	Nature of Compound	Mineral Species	Areas Where Found
		Cryolite ($2CaO.\ 2SIO_2.\ H_2O$) Xonotlite ($3CaC,\ 5SiO_2.\ H_2O$) Foshagite ($5CaO.\ 3SiO_2.\ 3H_2O$) Pectolite ($Na_2O.\ 4CaO.\ 6SiO_2$)	In boiler scales due to reaction of calcium hardness with silica
		Fayalite ($2FeO.\ SiO_2$) Mullite ($3Al_2O_3.\ 2SiO_2$) Olivine [2 (Mg. Fe) O. $5SiO_2$] Forsterite ($2MgO.\ SiO_2$)	On turbine parts due to dehydration of the correspond form
		Sodium silicates ($Na_2\ SiO_3$ and -$Na_2Si_2O_5$)	On turbine parts due to solubility in temperature steam
		Acmite ($Na_2O.\ Fe_2O_3.\ 4SiO_2$) Analcite ($Na_2O.\ Al_2O_3.\ 4SiO_2$ $2H_2O$) Natrolite ($Na_2O.\ Al_2O3.$ $3SiO_2.\ 2H_2O$) Neselite ($5Na_2O.\ 3Al_2O_3$ $6SiO_2.\ 2SO_3$)	In boiler sludge, boiler scale and turbine scale due to high temperature and more concentration of the components
6.	Sulphates of alkali and alkaline earth	Calcium sulphates ($CaSO_4$) Plaster of Paris ($CaSO_4.\ H_2O$) Gypsum ($CaSO_4.\ 2H_2O$)	On heat exchanger surfaces due to precipitation and dehydration
		Thenardite ($Na_2\ SO_4$ — V) Metathenardite ($Na_2\ So_4$ — I and III) Burkeite ($2Na_2SO_4,\ Na_2CO_3$)	On superheater and turbine blades due to precipitation on high temperature
7.	Sulphides of copper and iron	Chalcocite ($Cu_2\ S$) Covellite (CuS)	Copper tube heat exchanger due to presence of H_2S in water or sulphur getting reduced in the system
		Pyrrhotite (FeS) Troilite (FeS)	On boiler and super heater tubes due to high temperature reduction of sulphate or sulphite in the region
8.	Organic salts		On heat exchanger surfaces due to precipitation of organic material in water
		Calcium oxalate ($CaC_2O_4.\ H_2O$) or (CaC_2O_4)	In evaporator due to precipitation reaction of oxalic acid (if present) with calcium

4 METHODS OF SAMPLING

4.1 Mode of sampling shall be such that the deposits drawn are representative of the areas and location so that subsequent analysis is able to provide sufficient useful information regarding the mechanism of formation of deposits in those specific areas. Sufficient care shall be exercised while removing deposits from the metallic surfaces to avoid damage to the equipment and any metallic contamination in the deposits from the surface of constructional materials. If any deposit is extremely adherent on the surface and may neither be removed totally nor may be taken out alongwith the portion of metallic surface, a sample, even unrepresentative, may be drawn, even though it may not be suitable for all purposes.

The following techniques shall be adopted while drawing samples of deposits for analysis.

4.2 Collections

a) The sample collected from the point of formation shall be called 'gross sample', which will then be suitably prepared in the laboratory for final examination, which shall be called as 'sample for analysis'.

b) The deposits are to be examined visually on site before sampling 'gross sample'. Deposits with different appearances shall not be mixed together and a separate sampling has to be done. Separate sampling, areawise or different processes stagewise, is desirable.

c) Loosely adherent deposit on the surface (such as sludge, corrosion products, biological deposits, etc), may be drawn by a suitable scrapper like spatula, spoon, thin piece of wood, cardboard piece, chisel, sharp edged steel, scrapper including

knife or by any mechanical shock including the use of hammer.

d) Hard adherent deposits on the surface shall be removed by scrapping method with the aid of sharp pen, knife, cautiously and by keeping plain paper or suitable envelope below the surface for collecting the scrapped powder. Scrapping of the deposits shall be done layerwise (starting from upper most layer of the deposit), each time using the knife in such a way that the cutting edge of the knife is horizontal to the surface. This will avoid scratch on the metal surface while scrapping the last layer of the deposit. If a portion of the surface on which the deposit has been formed, may be taken out by cutting a small section of the pipeline or tube, then the deposits may be removed by applying mechanical or thermal shock to the piece.

e) If the deposits can not be removed in the original form by the above means then it is advisable to put the portion of the surface containing deposit into an inhibited acid (to avoid reaction with bare metal). This will help in disintegration of the deposits and a simple mechanical shock may ease their dislodging from the surface. Both soluble and insoluble portions of the deposit in acid may then be joined together and evaporated to dryness. Analysis of such a sample will indirectly predict the composition of the deposit. The nature of reaction with the acid used may, however, be noted immediately to correlate the findings of the analysis. Inhibited hydrochloric, hydrofluoric or a few organic acids may be usefully employed in the above process.

f) If the deposits are found on rough or irregular surfaces, pointed end of knife or needle shall be used to scrap the deposits or otherwise, mechanical or thermal shock shall also be applied. Deposits of loosely adherent nature may however be taken out by using iron brush and samples are collected as usual on paper sheet or in suitable envelopes.

g) If the deposits are in the form of slimes, it is always better to collect the samples before they get dried. The moist deposits or the deposits in the form of slurry, so dislodged shall be kept in sealed containers under refrigerated conditions for bacteriological examination. Subsequently, the samples can be preserved in the dried form for further examination.

4.3 Quantity

There is no specified quantity of deposits to be drawn as a sample for analysis. Depending upon the nature of tests to be carried out the quantity of sample may be decided by the analyst. As some deposits cannot be dislodged totally from the surface and individual samples has to be considered for different process stages to identify the problem of deposition. A sample as little as 0·1 g is usually sufficient to get sufficient informative data. About 5 g of deposit sample is desirable for an extensive X-ray diffraction study. Routine chemical analysis usually is possible with about 10 g of sample while elaborate investigations may require about 100 g sample.

4.4 After collection, the sample shall be kept in a suitable container properly sealed to prevent any contamination during the transport. For dry deposits, a glass or plastic container shall be adequate with proper sealing arrangement.

4.5 The sample after being sealed in a suitable container shall be labelled properly, with name and description of the equipment from which the sample was obtained, the precise location, the appearance and the precise extent of deposit prior to removal, method used in removing the sample, etc, and any other information noted during collection of the sample.

4.6 Laboratory Sampling

The 'gross sample' shall be first air dried properly. If the quantity is very less (less than 1 g) it is advisable to mix the identically appearing sample of the same area and make a composite. Part of the sample shall be kept as it is for reference and the rest shall be powdered suitably in an agate mortar, and mixed properly to make it thoroughly homogeneous. This shall then be treated as laboratory sample.

5 PRELIMINARY EXAMINATION OF DEPOSITS

Before removing the deposits from the surface, an assessment of the deposit thickness, area covered and approximate quantity shall be made and recorded. It is desirable to have photograph of the deposits, if possible, and compare the same with that of the bare surface of the metal. Then, the means of removal of deposits from the surface shall also be recorded with any other observation noted during dislodging the deposits. In case of mechnical removal, using knife or brush of any copper coating on the tube surface (below the deposits) may be noted and reported.

After the removal of the deposits, the deposits shall be examined for the following and recorded:

a) The physical appearance of the deposits, visual examination viz, size, shape colour, odour, crystalline or amorphous consistency, hardness, magnetic property, etc.

b) The texture of various layers and recognition of individual crystals if any under microscopic examination and compare with the standard compound which is suspected to be present in the deposit.

c) The solubility of the deposits on treating a portion with water and note down the *p*H of the water extract.

d) The possible form of compound present in the deposit, namely, organic matter, sulphur, etc, and the change in colour and odour on heating a portion of the sample.

e) The free water present on drying a portion of the deposits between 35 to 40°C.

f) The presence of oil on shaking a portion of the deposits with chloroform or solvent ether.

g) Observations, if any, on grinding a portion of deposits.

h) The presence of carbonates, iron sulphide, etc, on treating a portion of deposits with hydrochloric acid.

j) The major, minor or trace constituents on spectrographic, or petrographic, examination, if the facility available.

k) Loss in mass due to decomposition of organic and a few other inorganic C, N and S compounds.

6 LABORATORY SAMPLE PREPARATION

6.1 If sufficient information is available with the preliminary examination of the deposits, it would be possible to predict its likely composition. Further, when significant differences in deposit structure and behaviour are available with preliminary examination, it is desirable to divide the different portions to analyse separately. Separations are sometimes even made with a sharp blade, preferably under a high power lens. The different portions are taken, then kept in different bottles or even test tubes with proper markings.

6.2 It is further advisable to keep a portion of the deposits as a reference sample and for X-ray diffraction or absorption spectroscopy examination and the rest is ground in a mortar after getting rid of free water and oil from the deposits namely by drying at 35 to 40°C and washing by chloroform and ether respectively.

6.3 The oil free and dried material after grinding is quartered down to a sample of approximately 10 g (if so available, otherwise the whole quantity whatever is available) and is further ground to pass an IS Sieve of 150 micron [*see* IS 460 (Part 1) : 1978]. This is further bottled or tubed and properly capped after thorough mixing. The bottle or tube is further labelled with all necessary information. This laboratory sample is then ready for chemical analysis.

6.4 It is however, necessary to have special size sample for some particular tests for which a separate sampling shall be done from the gross sample after due crushing or grinding, whatever may be applicable. As for example, portions on which refraction index is required shall be crushed to finer size so that it is not too small for clear solution, whereas the portion shall be ground to 63 micron or finer for X-ray and spectrographic examination, 53 micron or finer and 150 micron or finer for microscopic examination. Various other physical and chemical treatments may be given to portions of the sample undergoing identification.

7 METHODS OF IDENTIFICATION OF DEPOSITS

7.1 The identification of water formed deposits does not involve using single method but of co-ordinating a number of methods to make each supplement to the other. No single method is available to tell all about a sample. The following types of analysis are made to identify the constituents of the deposits:

a) *Chemical Analysis*

The results of analysis are generally reported in terms of basic oxides and acid anhydrides to verify whether the sum of all the constituents approximates to about 100 or not. This procedure does not provide for reporting definite molecular combinations or it signifies that any of the oxides are present in the sample. However, probable compound can be interpreted if sufficient information is available with preliminary examination or any other selective tests applied.

b) *Microscopic Analysis*

Results of analysis are in the form of definite combinations but without indicating any amount. Under this heading, identification of individual crystals and amorphous lumps and also of micro organisms present, if any, are made.

c) *X-ray Diffraction Analysis*

The results give the identification of crystalline compounds present in the form of definite combinations.

d) *Spectroscopic Analysis*

The results give both qualitative and quantitative estimation of cationic constituents provided the sample has low volatile constituents.

8 CHEMICAL ANALYSIS

8.1 The following scheme of analysis with brief procedures of individual items outlined thereafter, shall be followed :

1 g Laboratory Sample of Deposit (Moisture and Oil Free)

Treated with Water, Boiled and Filtered

Filtrate

1. Add equal volume of hydrochloric acid
2. Precipitate, if any, filter otherwise evaporate the liquid portion in a porcelain basin to dryness on water bath
3. Heat for 1 h at 130°C
4. Add 1 : 1 hydrochloric acid (30 ml) and mix
5. Warm for 10 minutes on water bath

Residue

1. Take in platinum crucible
2. Dry
3. Ignite and cool
4. Mix with fusion mix six times (approximately) the mass of residue
5. Extract with hot water and transfer into porcelain basin
6. Add concentrated hydrochloric acid (30 ml)
7. Evaporate to dryness on water bath
8. Heat for 1 h at 130°C
9. Add 1 : 1 hydrochloric acid
10. Warm for 10 minutes in water bath

Filtrate

(A)

Mix the filtrate (A) and (B) if the water soluble portion of deposits are less; otherwise proceed the analysis individually

Residue

(B)

1. Take into a platinum crucible
2. Ignite and weigh
3. Add hydrochloric acid and little perchloric acid
4. Evaporate and fume out (80°C) and finally ignite for a minute (800°C) and weigh (loss in mass is due to water soluble silica)
5. Fuse the residue with sodium bicarbonate and extract with hot water. Mix it with the filtrate (A)

Filtrate

(B)

Residue

(Ignite and Weigh)

NOTES

1 Solution of water formed deposits is first tried with a mixture of mineral acids, including hydrochloric, nitric and perchloric acids, and the method of analysis is followed as per scheme outlined above, but without fusing the sample. In case, the deposits are partially soluble in the mineral acids, it is advisable to fuse the sample as outlined and proceeds for analysis. If the main part of deposits are soluble in acid, it is better to filter the sample and proceed for analysis, in two parts, one soluble portion and the other insoluble portion fused with fusion mixture.

2 During hydro fluorization (HF) use of little sulphuric acid may also be made in place of perchloric acid. Perchloric acid is however preferable which will not impart introduction of sulphate ion making some of the analysis interfered.

3 If a large quantity of phosphate is present in the deposit fuming the samples with mineral acids will separate phosphate alongwith the silica and some of the phosphates may be loss as phosphoric acid, if too much sulphuric or perchloric acid is used in hydro fluorization treatment to the silica residue.

4 The silica residue after, hydro fluorization treatment may also include barium sulphate and tin, as well as sodium and potassium which are however, returned to the original sample, after fusion.

5 The solution reserved for testing the different mixed oxides is divided into several aliquots, as to yield the best volume for each determination as per procedures given in **8.2**.

8.2 Procedures

8.2.1 *Water Soluble Portion and* pH

One g of laboratory sample is dissolved in 50 ml of distilled water. After shaking well, it is allowed to settle. A drop of clear water is then put on a *p*H paper with glass rod and the *p*H of water soluble portion is recorded. The *p*H of the distilled water used may also be recorded for comparison. The mixture shall be heated to 50 to 60°C, well stirred. It shall be then allowed to settle and cool. Then it is filtered in previously dried and weighed asbestos Gooch crucible or sintered glass Gooch crucible and washed properly. Finally the crucible is allowed to dry in an oven at 105 ± 5°C for an hour and weighed.

$$\text{Water soluble portion, percent by mass} = \frac{M_1 - M_2}{M_1 - M} \times 100$$

where

M = mass of empty crucible;

M_1 = mass of crucible before heating; and

M_2 = mass of crucible after heating.

NOTE — Normally the water-soluble content of a water formed deposit will be low, but presence of sodium salts of chloride, phosphate, silica, carbonate and bicarbonates along with calcium hydroxide, etc, shall not be ignored and tested for.

8.2.2 *Moisture*

One g of laboratory sample is taken in a clean porcelain crucible, previously heated up to 800°C, cooled in a desiccator and weighed. The sample alongwith the crucible is then kept in an oven maintained at 100 ± 5°C, for an hour. After taking out the crucible from oven, it is cooled in a desiccator and weighed again.

$$\text{Moisture content, percent by mass} = \frac{M_1 - M_2}{M_1 - M} \times 100$$

where

M = mass of empty crucible;

M_1 = mass of crucible with sample; and

M_2 = mass of crucible after heating.

8.2.3 *Loss of Ignition*

After weighing out the crucible for final mass M_2 in **8.2.2**, the crucible is kept in a furnace at 400 ± 50°C and then the temperature of furnace raised to 800°C, within half an hour. The temperature of 800 ± 25°C is then maintained for one full hour and then the crucible is taken out, cooled in a desiccator and weighed.

$$\text{Loss on ignition, percent by mass } L = \frac{M_2 - M_3}{M_2} \times 100$$

where

M_2 = mass of crucible as in **8.2.2**, and

M_3 = mass of crucible after ignition.

NOTES

1 While the moisture content of the sample will indicate the presence of certain hydrates in the deposits, ignition loss will indicate the presence of organic matter, carbon (carbonate, bicarbonate), sulphur (sulphite, sulphide) nitrogen (nitrate, ammonium salts), etc.

2 If the deposits are suspected to have considerable carbonaceous material including algae, fungi, etc, it is better to have a separate ignition loss test as above, but at 500°C, and the percentage loss shall be recorded. After this test, the total loss on ignition shall be determined at 800°C.

8.2.4 *Determination of Oil and Grease*

One g of laboratory sample is taken in an asbestos pulp thimble having sufficient porosity, but not allowing by any solid particles of sample to pass on. The sample in thimble is then covered with a small piece of cotton fibre and the thimble is put into Soxhlet distillation apparatus, with about 50 ml of benzol (1 : 1 ratio of benzene and alcohol) in the Soxhlet flash of 100 ml capacity. After circulating cold water into the condenser, the Soxhlet flask is heated on a water bath for about an hour.

On completion of the extraction, the thimble is taken out and dried in an oven at 100 ± 5°C for 2 h and weighed. (If presence of ammonium nitrate is suspected in the sample, it is better to dry at 70°C.) The loss in mass of sample in the thimble represents all benzol soluble material including oil and the moisture in the original sample.

NOTES

1 In water formed deposits, generally the 'benzol' soluble portion is oil, grease, etc. Though benzol may dissolve sulphur, resins, some specific organic compounds also, the presence of these is very unlikely in the deposit.

2 Presence of oil in the deposits is indicated where even after drying, the sample smears or agglomerates, during its pulverization.

8.2.5 *Carbonates and Bicarbonates*

One g of laboratory sample is taken in a previously weighed Schroetter's apparatus and fitted with the drier column properly filled with 1 : 1 hydrochloric acid in the thistle portion. Then hydrochloric acid is allowed to fall into the sample chamber by opening the stopcock of the thistle portion very slowly. Finally when all the acid is in contact with the sample, the Schroetter apparatus is kept on the surface of 40 to 50°C for 5 minutes and then finally

weighed. The loss in mass of Schroetter's apparatus gives the mass of carbon dioxide escaped from the sample which may be due to the presence of carbonates and bicarbonates.

NOTE — A qualitative test for carbon dioxide shall be first made, by boiling the sample mixture with water and then by adding mineral acid to the mixture and then passing the evolved gas in both cases in lime water. If, in both cases the result is positive, presence of both bicarbonates and carbonates are indicated. In case, both are present, carbon dioxide evolved by heating the sample water mixture is absorbed in soda lime bulb and bicarbonate is calculated out. However, chances of presence of both bicarbonate and carbonate in deposit sample are rare.

8.2.6 *Sulphides*

One g of laboratory sample is taken in a 250-ml conical flask with an inlet tube reaching nearly the bottom. The outlet tube from the flask is connected to two absorption bulbs containing lead acetate solution and previously weighed. In the flask 10 ml concentrated hydrochloric acid and 20 ml ether are placed and a current of pure carbon dioxide is passed. The generating flask is slightly heated to ensure complete decomposition. By the difference in mass of the bulbs before and after the experiment, the hydrogen sulphide content absorbed shall be known and using this hydrogen sulphide, sulphur is calculated.

NOTE — A qualitative test for hydrogen sulphide using sulphuric acid and lead test paper may first be made before undertaking the above estimation.

8.2.7 *Alkalis*

Alkalis are determined by flame photometric method as given in IS 1917 : 1962 after fuming a known mass of the sample to near dryness with hydrofluoric acid and perchloric acid, dissolving the residue in dilute hydrochloric acid and making up to a definite volume.

NOTE — Alkali salts usually are found with the water soluble portions of the deposits, but in some cases they may be water insoluble also when they occur in complex compound form. Hence determination for alkalis, both water soluble and total, is advisable to account for the nature of the deposit.

8.2.8 *Ammonia*

Ammonia as total nitrogen is determined by Kjeldahl method, as prescribed in IS 5194: 1969. One g of laboratory sample is placed in the distillation flask with splash bulb as described in the modified Kjeldahl procedure for organic substances and the material decomposed with ammonia free caustic solution. The ammonia is distilled into an excess of standard acid and the ammonia content is determined as usual by titration of the excess of acid.

NOTE — If ammonia is present in the deposit, some of ammonium salts would always be present in water soluble form. Hence it is advisable to have spot test for presence of ammonia before undertaking the above laborious test. Spot test for ammonia is conducted by heating the deposit with a solution of sodium or calcium hydroxide and smelling the evolved gas for the characteristic odour or seeing the effect of the gas on mercuric chloride, moistened filter paper which would turn black, if ammonia is present.

8.2.9 *Phosphate*

One g of the laboratory sample is placed in a porcelain dish and is digested for an hour with concentrated nitric acid, the dish being covered by a watch glass placed on a steam bath. The acid is then diluted with half of its volume of water and the solution is filtered into a porcelain dish of sufficient capacity to hold the filtrate and washings (1 : 1 nitric acid). The filterate and washings are then evaporated to dryness. Meanwhile the insoluble residue with filter paper are ignited in a platinum crucible and the residue fused with ten times its mass of sodium carbonate. The fused mass is extracted first with water and then with 1 : 1 nitric acid and this solution is added to the main solution. The combined solutions are then evaporated to dryness and baked to dehydrate the silica at 130°C. The residue is taken up with a few ml of nitric acid, the solution diluted, filtered and silica washed with dilute nitric acid solution. The filtrate is then taken for estimating phosphate either gravimetrically (magnesium ammonium phosphate) or colorimetrically (molybdenum blue) as per IS 6361 : 1971.

NOTE — If a large amount of phosphate is present in the deposit sample, some of the phosphate may be separated with the silica and loss as phosphoric acid, during fuming with hydrofluoric acid, sulphuric acid/perchloric acids. Hence, it is advisable to conduct examination and estimation of phosphate on the original deposit sample, rather than in the alkali extract of the sample after fusion with sodium carbonate/fusion mixture.

9 ANALYSIS OF FINAL FILTRATE AFTER SILICA DETERMINATION

9.1 The final filtrate from the silica determination kept reserved for analysis (as under the scheme of treatment of main analytical sample) is made up to 1 litre in a volumetric flask. From this, suitable aliquots are drawn for estimating different contents as below. The size of the aliquots shall however be based on preliminary examinations so as to yield the best volume for each determination.

9.1.1 *Sulphate*

A suitable aliquot is taken and the hydrochloric acid concentration is adjusted to 2 to 5

percent (*v/v*) and the aliquot is boiled. While boiling, 10 ml of 10 percent barium chloride is added to it and the boiling is continued for 5 more minutes. The container is then kept on water bath till precipitate settles. It is then filtered and washed with 0·1 percent hydrochloric acid solution. The precipitate is dried, ignited and weighed as barium sulphate, from which the mass of sulphate is calculated by multiplying the mass of barium sulphate by 0·342 97.

9.1.2 *Manganese*

To a suitable aliquot, sufficient quantity of sulphuric acid with little ammonium sulphate is added and the solution is first boiled for a few minutes and then evaporated to dryness and further to fumes of sulphite. The residue is then brought into a solution containing atleast 10 to 15 ml of concentrated sulphuric acid and 5 to 10 ml of concentrated phosphoric acid per 100 ml. Then 0·2 to 0·4 g of potassium iodate is added and the solution is boiled for a minute, kept hot for 5 to 10 minutes, cooled, diluted to a proper volume and compared with a solution of known manganese content, prepared in a similar manner. Not more than 1 mg of manganese per 50 ml shall be present at the time of comparison.

9.1.3 *Titanium*

The separation of titanium from molybdenum, phosphorus and vanadium is made by carefully neutralizing the greater part of the free acid present by addition of a solution of sodium hydroxide and pouring the mixture into 150 ml of a hot, normal solution of sodium hydroxide filtering the washing with hot dilute sodium hydroxide (0·5 N). It is advisable to repeat the precipitation to remove traces of the interfering elements. The titanium is then brought into solution with sulphuric acid. Hydrogen peroxide is then added to it, and the colour produced is compared with standard colour obtained from standard solution of titanium. Colour comparison can best be made on samples containing 0·05 to 5 mg of the element, as larger amounts produce too deep a colour for accurate comparison. Further aliquot to be taken shall not have more than 4 percent iron which would also produce colour. However, 0·1 g ferric oxide in 100 ml solution = 0·2 g of titanium oxide shall be allowed.

9.1.4 *Chromium*

A suitable aliquot containing not more than 0·17 g chromium (as chromate) is boiled with hydrochloric acid to expel oxidizing reagents from the solution. 5 ml of 1 : 1 hydrochloric acid is then added followed by a known amount of standard 0·1 N ferrous ammonium sulphate till the solution changes from yellow through olive green to deep grass green. For every 0·1 g chromium about 65 to 70 ml of 0·1 N ferrous salt solution shall be added. After 5 minutes, the excess of this reducing reagent is back titrated with dichromate in presence of diphenylamine indicator.

$$\text{Mass in g of chromium in the aliquot} = 0{\cdot}001\,734 \times A$$

where

A = ferrous ammonium sulphate in ml consumed

9.1.5 *Vanadium*

To a suitable aliquot, cooled to 15 to 20°C, add approximately 0·1 N potassium permanganate solution until very strong pink colour remains for a minute (an excess permanganate does not harm). Then the vanadium is reduced by adding approximately 0·1 N ferrous ammonium sulphate until a drop of solution on spot plate to a drop of 0·1 percent solution of potassium ferricyanide results in immediate formation of a blue colour (presence of excess of ferrous iron). 3 to 5 ml ferrous ammonium sulphate is then added in excess and the solution is stirred for 1 minute. The excess ferrous ammonium sulphate is then oxidized with 15 ml of 10 percent freshly prepared ammonium persulphate and the solution is stirred vigorously for one minute. The solution is then titrated with 0·1 N permanganate solution with constant stirring until a faint pink colour appears, persisting for one minute. Care shall be taken that all along the temperature of the solution is kept between 15 to 20°C and a blank determination is also carried out in the same way.

$$\text{Mass in g of vanadium in the aliquot} = 0{\cdot}005\,096 \times A$$

where

A = volume in ml of potassium permanganate solution consumed less blank.

9.1.6 *Calcium, Magnesium and Barium*

With a suitable aliquot taken, calcium and magnesium are determined in series, as already given. To the solution taken, a few ml of concentrated nitric acid is added and boiled. After cooling, 2 g of ammonium chloride is added and again boiled. The solution is then neutralized with ammonia and slight excess ammonia is added. After settling hot for a few minutes, filter the solution through Whatman No. 30 filter paper, where iron and aluminum are removed as residue. The filtrate is again made slightly excess alkaline with ammonia

and boiled. While boiling saturated ammonium oxalate solution is added till complete precipitation and slightly in excess. The precipitate of calcium oxalated is allowed to settle for 30 minutes and then filtered hot through Whatman No. 30 filter paper. After redissolving the precipitate in hydrochloric acid again, the calcium oxalate is reprecipitated and filtered and washed thoroughly with hot water. The residue is then dried, ignited, and weighed as calcium oxide.

9.1.6.1 To the filtrate, properly cooled to room temperature, excess ammonia is added followed by 10 to 15 ml ammonium hypophosphate and the solution is vigorously agitated with glass rod for a few minutes and then kept over night. It is then filtered cold through Whatman No. 40 filter paper and the residue washed thoroughly with ammoniated water. The residue is then dried, ignited and weighed as combined mass of manganese and magnesium. This residue is then dissolved in hot concentrated nitric acid. A few ml of concentrated sulphuric acid is added and fumed. Manganese content is then determined as given in **9.1.2** and the amount of magnesium from the above residue is calculated by difference.

9.1.6.2 To another aliquot, about 200 ml of water is added to make the solution slightly acidic (about 1 to 2 percent) and is heated to boiling. Then a slight excess of hot dilute sulphuric acid is added. The precipitate is allowed to settle and then filtered through Gooch crucible and washed twice with dilute sulphuric acid (0·5 percent) and finally with hot water till free from acid. The precipitate is dried and weighed as barium sulphate.

NOTES

1 Double precipitation of calcium reduces occlusions to significant proportion of the interfering radicals of the group, while the phosphate of copper, nickel and zinc will largely remain soluble in ammoniacal solution though they are insoluble in neutral medium.

2 If more accuracy in magnesium determination is required or if the original deposit contains very large amounts of copper, nickel, zinc and lead it is better to remove the interferences by passing hydrogen sulphide and filtering off the sulphide precipitate but the metallic constituents usually present in water formed deposits do not warrant this.

9.1.7 *Lead, Copper, Nickel, Zinc, Iron, Tin, Aluminium*

9.1.7.1 From a suitable aliquot taken, copper and lead are first removed and determined, phosphate is eliminated and then the rest of the constituents are determined as per the method of analysis already stated.

9.1.7.2 The acidic solution containing 2 to 3 ml of free concentrated nitric acid is taken and warmed to 50 to 60°C and electrolyzed with a current of 1 ampere and 2 to 2·5 volts. The progress of electrolysis is watched and the cathode is removed as soon as completion of deposition is detected by evolution of gas on its surface. The completion of action shall be ascertained with by addition of water to the electrolyte and observing whether the newly exposed surface of the cathode remains bright. Then the detachment of the cathode is preceeded by removal of the electrolyte and simultaneously washing the cathode without interrupting the current. The cathode after detachment is washed properly with water and then with alcohol and dried and weighed. The amount of copper and lead deposit is then known by difference in mass of the bare cathode. This deposit is then redissolved in nitric acid and replated where copper remains as deposit and the lead remains in solution.

This solution is then evaporated to dryness and fumed with sulphuric acid. After cooling, 1 : 6 hydrazine sulphuric acid is added and the solution is boiled, filtered into a Gooch crucible washed with dilute sulphuric acid to remove soluble impurities. The residue is finally washed with 50 percent alcohol solution till free acid is removed. The residue after drying is weighed as lead sulphate, from which the amount of lead is calculated by multiplying with 0·683 3.

9.1.7.3 The main mother liquor obtained after first plating of copper and lead together is then divided in two parts:

a) To the first part, nitric acid is added to adjust the concentration as 5 ml of concentrated acid for every 100 ml solution. The solution is then warmed to 45°C and ammonium molybdate reagent is added to it with constant stirring till complete precipitation.

   It is then allowed to settle for an hour, filtered through asbestos Gooch crucible. The residue is washed with 1 percent potassium nitrate and discarded.

   The filtrate together with washing is boiled, made alkaline with ammonia, settled, warmed and filtered. After discarding the filtrate, the residue is tin and aluminium.

   The residue properly washed with hot water, is dissolved in dilute hydrochloric acid and boiled till clear solution is obtained. The acidity is maintained at 5 to 10 ml acid per 200 to 500 ml solution.

The solution is then chilled and an excess of 10 percent freshly prepared cupferron solution is slowly added with stirring. When the precipitate becomes brittle after 20 to 30 minutes, it is filtered and the residue washed speedily with cold water. The precipitate is then dried and ignited very carefully and slowly at low temperature until filter paper is consumed and precipitate is converted to stannous oxide in which form it is weighed after strong ignition.

The mass of tin = Mass of stannous oxide × 0·787 7

The filtrate after filtering the tin residue is dried and fumed with sulphuric and nitric acids. After getting cooled, 1 : 1 hydrochloric acid is added to dissolve the residue. To the solution, a few millilitre of concentrated nitric acid is added and boiled to expel nitrous fumes. The solution is then made alkaline with ammonia maintaining some excess ammonia and boiled. It is then allowed to settle warm for a few minutes and filtered hot through Whatman No. 31 filter paper. The residue is washed with 1 percent ammonium nitrate solution and finally with hot water till it is free from soluble impurities, dried, ignited and weighed as aluminium oxide quickly (as it will absorb moisture to gain in mass).

NOTES

1 Only freshly prepared solution of cupferron shall be used, as it decomposes slowly. Stability of cupferron is sometimes enhanced by adding 50 mg of acctophenetidide per litre of 6 percent aqueous solution. It is also better to suspend a small bag containing ammonium carbonate in the bottle of dry cupferron to maintain its quality for larger period.

2 Iron-cupferron precipitate is only slowly attacked in the cold by 2 N hydrochloric acid, but hot acid decomposes it. For this reason, the precipitation must be done in the cold.

3 Nitric acid shall be absent when cupferron is used as a precipitant.

4 If the mixed oxides precipitate is bulky, it is advisable to oxidize it with nitric and sulphuric acid and then reprecipitated with ammonia.

5 Large amounts of aluminium may be determined by sodium fluoride, sulphuric acid filtration using phenolphthalein indicator.

b) The other part of the mother liquor is boiled to expel nitrous acid fumes and then made alkaline with ammonia while it is hot. Allowed to settle for a few minutes and filtered through Whatman No. 31 filter paper, while it is hot. The residue is dissolved in 1 : 1 hydrochloric acid and boiled. While boiling it is reduced by freshly prepared stannous chloride solution, till yellow colour is destroyed and a few drops are then added in excess. It is then cooled, about 10 ml of saturated mercuric chloride solution is added, and the mixture shaken. Then it is titrated against standard potassium dichromate in presence of diphenylamine indicator.

1 ml of 0.1 N potassium dichromate = 0·005 584 g iron

The filtrate is acidified with sulphuric acid to about 0·02 N concentration. Hydrogen sulphide is then passed to it rapidly under pressure. The precipitate is then filtered, washed thoroughly with cold water, and ignited and weighed as zinc oxide.

Mass of zinc, mg = Mass of zinc oxide (Zno) × 0·803 4

After filtering of zinc precipitate, the filtrate is boiled to remove hydrogen sulphide and cooled. One to two g of tartaric acid is added to prevent the precipitation of hydroxides of iron, aluminium and chromium, etc, and 5 to 10 ml of 10 percent solution of ammonium chloride is added to keep manganese in solution. Ammonium hydroxide is then added till the solution is slightly alkaline. It is then heated to nearly boiling and the alcoholic solution of dimethylglyoxime is added until reagent is approximately seven times the mass of nickel present in the sample. Ammonium hydroxide is now added until the solution has a distinct odour of this reagent. The precipitation of the scarlet red nickel salt is then hastened by stirring. The mixture is kept on steam bath for 15 to 20 minutes and filtered off in a Gooch crucible and the residue washed with cold water and dried for about 2 hours at 110 to 120°C and weighed as ($C_6H_{14}N_4O_4Ni$) :

Mass of nickel, mg = mass of precipitate × 0·2031

10 MICROSCOPIC ANALYSIS

10.0 The aim of this analysis is to magnify the deposits under observation, so that their distinctive form and features become recognizable.

10.1 Biological Examination

For this the deposit is taken with its environmental water, properly refrigerated and preserved. If the sample can not be preserved properly, it is advisable to kill the bacteria by adding suitable bactericide to the sample and then examined under microscope. In no case, the biological slime shall be allowed to dry, otherwise identification would be difficult.

10.1.1 A small drop of the sample bearing liquid is put between a glass slide and a cover glass, and kept, under microscope, to examine shape, colour or characteristic reaction to some reagents. The image of the micro organisms is magnified as the light continues upward through the lenses of the objective and the eye piece. The magnified image is either observed directly at the eye piece or photographed with a camera and compared with standard micro organisms behaviour towards their living functions, metabolic by-products and skeletons of dead organisms.

10.1.2 Recognition by shape is sometimes difficult and for confirmatory information, the organisms may be inoculated into a culture medium. After a suitable incubation period, the medium is tested for specific metabolic by-products of the organism. For example, the formation of hydrogen sulphide in a sulphate culture medium is confirmatory evidence of the presence of sulphate reducing bacteria.

10.1.3 Similarly, when the organisms are covered with iron oxide sheaths, the sheath is first dissolved in dilute acids, the dissolved iron will colour the liquid between the cover glass and slide indicating the presence of an iron bacteria.

10.1.4 Some organisms will absorb specific dyes and be recognizable by this property also.

NOTES

1 A microscope with three objectives (X 10, X 43 and oil immersion X 97) two eye pieces (X 5 and X 10) and an Abbe condenser, assembled with an illuminator and mechanical stage is most suitable for biological examination.

2 Microscopic examination generally gives a qualitative idea of presence of organic matter including bacteria. Quantitative values are assigned only when specific cultures can be made.

3 Samples containing too much clay or other suspended matter may first be separated from these before microscopic examination by suitable decantation and filtration. When the number of organisms is so small that a single drop of liquid might not contain any observable species, the sample should be centrifuged to concentrate the organism and the observations should be made on the concentrate.

4 If on heating, an original dried sample of deposit gives pronounced darkening of the sample on heating, it gives an indication of presence of organic matter. The odour of the burnt compound may also be used to identify many types of organic material such as carbohydrates, proteins, amines, oil, coal, sulphur compounds. This may further be verified by extracting the sample with suitable solvent and using the microscopy, X-ray diffraction or molecular absorption spectroscopy to confirm the initial suspicion.

10.2 Petrographic Examination

A pinch of powdered sample preferably 150 micron size or finer supported by a glass slide is kept on the chemical microscope and the image of the object is magnified as the light passes upward through the objective. The magnified structures, which are generally constituted of more than one species are compared individually with known structures of certain ions or species. The examination thus gives qualitative picture permitting distinction between several ions like phosphate, ferrous and ferric iron, copper, zinc, nickel, calcium, magnesium, manganese, sodium, sulphate, chloride and others and also furnish the information of the presence of specified compounds and elements in the deposit.

10.2.1 To estimate further the amount of certain species like amorphous material and glasses, examination is accomplished under a chemical microscope equipped with polarized light and the resolution noted.

10.2.2 Identification of the species in the deposit is also accomplished by immersion of the powder in oils of various refractive indices and then determination of the indices.

NOTE — The chemical microscope is a standard instrument and differs from the biological microscope in that, it is equipped with a graduated rotating stage and polarizing prisms. Light from a source is reflected by the mirror and passes through the polarizer and condenser. The polarized light passes through the sample and the image of the object, is magnified as the light passes through the objective, the analyser and the cross hair eye piece.

11 X-RAY DIFFRACTION EXAMINATION

The finely grounded sample enclosed in an air tight capsule or a suitable piece of large sample in flat form as required, is put into the diffractometer where the pencil of X-rays is intercepted by the sample. The position of the peaks on the recorder chart is then determined for any given type of X-radiation, by the distances between layers of atoms in the crystalline components of the sample. These interatomic distances are then calculated from the measured position of the peaks and used to identify the crystalline compounds of a sample.

11.1 To determine the compound quantitatively, use of detection of X-ray intensity is made by employing counter tubes in the diff-

ractometer, where a better integration of the overall composition of the sample is possible and sufficient patterns are almost quantitatively available within 30 minutes to 1 h.

NOTE — Accurate quantitative analysis depends on careful consideration of the absorption characteristics of the sample and how the sample is prepared. The minimum amount that can be detected may vary from 1 to 30 percent depending upon the efficiency of the compound as a diffractor and the absorption characteristics of other materials in the mixture. For example, 1 percent copper may be detected in magnesium hydroxide whereas 5 to 10 percent copper shall be present for detection in an iron oxide sample. Further, the different sample preparation techniques may have preferred orientations to increase the intensity of the strongest line, which although is acceptable for qualitative analysis (to detect even smaller amounts), but marks the other lines for quantitative analysis.

X-ray diffraction analysis may also be made with photographic method by taking pattern of diffracted lines on photographic film which however, takes much time, but giving pattern of high quality lines on a very small amount of sample (2 to 3 mg of finely ground sample only). However, the report of an X-ray diffraction analysis should give the name and formula for the crystalline species identified together with an estimate of the relative amounts of the identified phases present, and any remarks regarding crystal size, solid solution or other pertinent information that may have been obtained.

12 SPECTROSCOPIC ANALYSIS

12.1 Atomic Absorption

The finely ground sample in the form of solution is atomized into the wide vaporizing oxyacetylene flame provided with the atomic absorption spectrophotometer and the absorbance of the radiation is measured by the photometer in reference to a calibration curve showing the relationship of absorbance to concentration. As the equipment has relative freedom of interference of one element from another, all elements present in the sample of the deposit may be easily determined one by one separately, and quantitatively.

NOTES

1 The principle of measurement is that when atoms of an element are diffused by atomization into a flame, they absorb light of the same wave length as is emitted at high temperature by that element. The amount of light absorbed by the atomized atoms is proportional to the element concentration in the vapour of the sample flame.

2 By noting down the absorption levels at a given wave length over a given time, the amount of element present is determined generally by integrating the area under the various peaks observed u sing a recorder for a given time period in which the sample was atomized.

12.2 Molecular Absorption

The sample is ground briefly with powdered potassium bromide and the mixture is placed in an evacuable die. A mechanical vacuum pump removes the entrapped air, and the mixture is then pressed at about 1 000 kg/cm^2. The pellet is then kept into the sample socket of molecular absorption spectrophotometer where the light from the source of infrared radiation (the filament of which operates at a low temperature) is passed through the sample, and absorption bands in the infra-region of the spectrum is noted that is in the wave length range from about 2 to 15 micrometer. Each absorption band will be characteristic of a particular grouping of organic substances which may be compared with the known sample.

NOTES

1 The method detects the presence ef organic compounds with their nature and grouping, but does not measure the compound quantitatively.

2 Detection of oil in turbine deposits and the type classification determination of the type and degree of degradation of amines, and possible identification of the source of stream pollutants may be made by molecular absorption analysis.

3 The temperature of the source must be carefully controlled to prevent change in the distribution of spectral intensity.

4 The principle of operation of the equipment is such that the source light after passing through the sample is broken up into various wavelengths by a prism or diffraction grating and after isolation of the desired wavelength band by a variable slit, the radiation falls on a detecting and measuring device most commonly using lead sulphide photocells. The output of the detecting device is then further amplified and fed to a recorder.